Measurement of Oil and Water in Seeds according to ISO 10565

Application Note 03

Summary

- Fast, accurate and repeatable
- Minimal sample preparation
- Simple linear calibration, no chemometrics
- Easiest, most reliable technique available; suitable for non technical personnel

Application

Accurate and fast determination of oil and water content in oilseeds is important to breeders, growers and buyers for determining the commercial value of crops such as rape (canola), sunflower, linseed (flax), soya bean and groundnut. Nuclear Magnetic Resonance (NMR) offers a clean, rapid and accurate alternative to traditional wet chemical techniques. Furthermore, calibrations are much easier to maintain than for Near Infra-Red (NIR). NMR is also used for measuring oil yield in the development of improved crop varieties and by seed crushers to measure oil content of the press cake and residues to determine extraction efficiency (ISO 10632).

Advantages of NMR

Solvent extraction techniques are commonly used for determination of oil content. However, these methods can be time consuming, require skilled operators and the use of hazardous solvents.

NIR is rapid but only measures the surface layer, so sample grinding may be required for improved reproducibility. In addition, NIR requires calibration using a large number of reference samples that represent a variety of factors including oil and water content, seed or grain type, particle size or colour. Therefore, the results may unknowingly be inaccurate if the sample is outside the range/scope of the calibration.

In contrast:

- NMR does not require solvents which are hazardous to use and in its disposal
- NMR is a bulk measurement technique and so measures all the oil in the sample regardless of the particle size
- NMR is the method of choice for sunflower seeds since it is unaffected by colour; as such it is used as an official USDA GIPSA method
- Oil and water calibrations may be obtained using just three samples
- NMR provides a rapid measurement for a variety of sample sizes
- NMR is used in the ISO 10565 method, which has been adopted by FOSFA (Federation of Oils, Seeds and Fats Associations).

The **MQC** benchtop Nuclear Magnetic Resonance (NMR) analyser provides an alternative method to wet chemistry and NIR; it is quick and easy to perform, simple to calibrate, and requires minimal sample preparation. As such it is ideal for routine operation without any requirement for additional chemicals, complicated calibrations or specialist operator training.

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Method

The oil and water measurement involves differentiating the two analytes on the basis of their NMR relaxation times. The NMR signal from solids within a sample decay rapidly, leaving signals originating only from oil and bound water. Subsequently the signal from the bound water decays leaving that from the oil only. The water is determined by taking the difference between the oil and combined oil + bound water signals. The ISO 10565 method recommends that the water content be less than 10 % for all seeds to obtain reliable results. In practice, this value may be a few percent higher depending on the seed type.

Calibration

As NMR is a comparative technique, a set of calibration standards consisting of real seeds with known oil and water contents must be obtained before measurements can take place. Although only three well characterised seed standards are required, it is recommended that at least six should be used, with oil and water contents that span the concentrations of interest. Oil and water reference values are normally determined using Soxhlet extraction and oven drying respectively.

The quality of the calibration will always be dependent on the accuracy of this reference data. However, NMR is more reproducible than wet chemical methods therefore errors are reduced by analysing more reference samples. Alternatively, a primary calibration for total oil content can be produced by NMR using the pure oil to be analysed.

Measurement

Samples are poured into glass NMR tubes up to a predefined mark and weighed. Large samples are normally conditioned at room temperature in a stable environment. Measurement time is typically 16 seconds per sample as per the ISO standard methods.

Results

Normally best accuracy is achieved when the standards are of the same species as the measurement samples. However, various seed types with oils that have a similar hydrogen density may lie on the same calibration.

Nine independently-analysed rape seed samples were measured. Their oil content varied from 39% to 51% and their water content varied from 5.2% to 7.1%. Calibrations for oil and water were developed according to ISO 10565 using Oxford Instruments' **MultiQuant** software, which allows simultaneous measurement of up to four sample parameters. In addition to oil and water content, it is also possible to quote oil per dry weight and normalised to a particular water content (for example 8 or 9%). The resulting calibrations are shown in Figures 1 and 2.

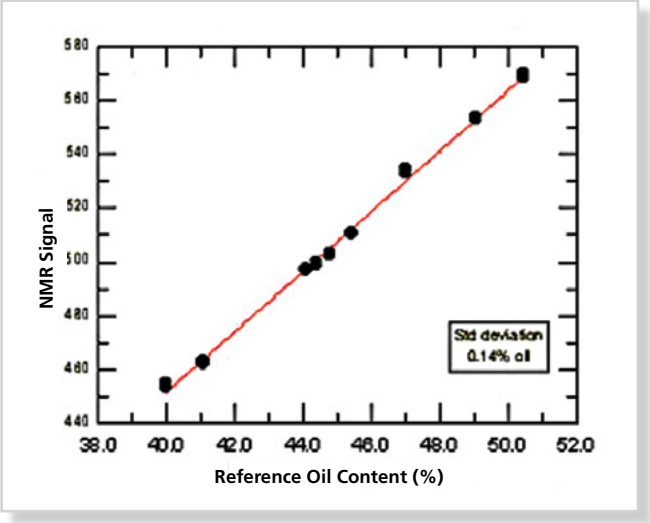

Figure 1: *Calibration of oil content of rape seeds in the presence of water (Standard deviation 0.14%)*

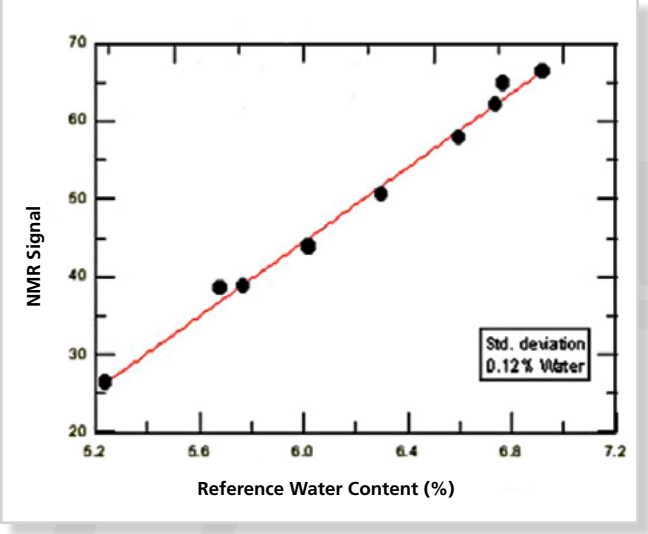

Figure 2: *Calibration for water content of rape seeds in the presence of oil (Standard deviation 0.12%)*

Instrument repeatability for oil was then tested by measuring one sample ten times without removing it from the instrument. Sample reproducibility was tested for oil content by measuring five different portions of the same sample. Instrument repeatability was shown to be 0.03% and sample reproducibility of 0.24% owing to the varying distribution of seed oil contents in each portion. The results from both sets of experiments are shown in Table 1.

value	Repeat Measurements										Mean	SD
44.25	44.29	44.25	44.22	44.23	44.26	44.27	44.22	44.23	44.22	44.18	44.24	0.03
value	**Portion Measurements**										**Mean**	**SD**
39.5	39.7		39.2		39.4		39.5		39.8		39.5	0.24

Table 1 Instrument repeatability and sample reproducibility.

Conclusion

- NMR is very stable over the long term and rarely needs calibration adjustment.
- NMR is insensitive to the air voids between the grains or powder.
- Measurement precision is good compared to wet chemical methods.
- Sample measurement time is rapid (typically 16 seconds)
- The NMR technique is non-destructive so the same sample may be measured multiple times before being analysed by other techniques.

In addition the **MQC** has the following advantages:

- The probes are easily removed (without tools) for changing probe size or cleaning.
- The auto-weighing feature used on the **MQC**5 increases sample throughput. Samples can be measured without the need to touch the keyboard or mouse, saving time.
- Automatic post-measurement calculation of oil content with respect to dry weight, 9%, or other water content reduces work.
- The ability to import and export data enabling the instrument to fit in existing systems.

Complete Package

Oxford Instruments offers various packages especially tailored to the measurement of oil and water in oilseeds and their residues (pressed cake or meals). There are two instruments suitable for this application both of which conform to the industry standard ISO 10565 for a range of sample volumes (given in brackets):

For large sample/seed analysis

- **MQC**5 with 51 mm (80 ml), or 40 mm (40 ml) diameter probes.

For small, low quantity or single seed analysis

- **MQC**23 with 26 mm (14 ml), 18 mm (7 ml) or 10 mm (1 ml) diameter probes.

The **MQC**5 (or **MQC**23) which can be controlled using its own built-in computer using Microsoft Windows® or via a stand-alone PC, also includes:

- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Oil and Water in Seeds' application
- Test/tuning sample
- Glass tubes
- Oil and water "calibration maintenance standards" (for 51 mm, 40 mm and 26 mm probes only)
- User manuals
- Method sheet

In addition you may also wish to purchase:

- Required: A precision balance (2 decimal places for 80 ml and 40 ml samples, and 3 decimal places for 14 ml, 7 ml and 1 ml samples)
- Optional: a dry heater and aluminium block with holes for sample conditioning at 40°C (26, 18 and 10mm probes only)

N.B. The ISO method specifies measurement at a nominal room temperature of 17°C-28°C. Conditioning at 40°C is preferable where precision measurements are required for oil content only.

Analyser

Electronics with integral PC

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